

**SIZE-CONTROLLED THE GOLD NANOPARTICLES
ON SILICON SUBOXIDE FILM GROWN BY
VAPOR DEPOSITION TECHNIQUES**

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ABSTRACT

In this work, size-control of the gold nanoparticles (Au NPs) on silicon suboxide (SiO_x) film grown by vapor deposition techniques was studied. Investigation of the effects of surrounding medium on the structural properties of Au NPs was studied first. RF magnetron co-sputtering technique was used to prepare “supported ON” and “embedded IN” structure of Au/ SiO_x films. As-prepared samples were annealed at different temperatures from 400 to 1000 °C to study the effect of annealing temperature on the growth of Au NPs. For both structures of Au/ SiO_x film, size and shape of Au NPs were observed to be temperature-dependent. Au NPs embedded IN SiO_x film seemed to appear and distribute uniformly on the surface of SiO_x film at 800 °C, while, Au NPs deposited on the surface of SiO_x film demonstrated a uniform dispersion of elongated and island-like Au particles at 400 °C. The shape of particles changed to spherical-like as annealing temperature gradually increases from 400 to 800 °C. Growth of closely packed SiO_x nanowires (NWs), which follows the solid-liquid-solid (SLS) growth mechanism, was observed at 1000 °C. Position and width of surface plasmon resonance (SPR) peak were greatly dependent on the size and shape of Au NPs. For the second part of this work, only Au/ SiO_x film with “supported ON” structure was studied due to its intriguing morphological properties besides simple controlled synthesis method. Homebuilt plasma-enhanced chemical vapor deposition (PECVD) technique and direct current (DC) sputter coater were introduced to prepare Au on SiO_x film. Effect of $\text{N}_2\text{O}/\text{SiH}_4$ flow rate ratio on growth of Au NPs was studied with annealing temperature being kept constant at 800 °C. High concentrations of Au NPs were distributed evenly on the surface of SiO_x film prepared at optimal flow rate ratio of 30. Annealing process improved the SPR peak for all as-deposited samples. FWHM of SPR peak was the dominant factor to correlate with the size of Au particle. In order to reduce unnecessary contamination that may occurred during the removal of sample from vacuum chamber to sputter coater, a one-step process was introduced - Hot Wire Assisted PECVD technique. Evaporation of Au controlled by a shutter was carried out in a vacuum chamber immediately after the deposition of SiO_x film at optimal flow rate ratio of 30. Role of SiO_x film as a barrier for preventing agglomeration of Au NPs was studied. It was confirmed that the weak Au- SiO_x chemical interaction allows the precise study of Au NPs alone by eliminating other unwanted factors. Lastly, the effect of substrate heating on the growth of Au NPs was investigated to determine the possibility of preparing a uniform and smaller size of Au NPs without using the post thermal-annealing technique. It was found that Au NPs with uniform size around 6.45 nm can be obtained as substrate temperature is set at 300 °C. No formation of NPs can be observed below 300 °C. Further thermal annealing process at lower temperature of 200 °C was shown to improve the distribution of Au NPs. FWHM of SPR peak became narrowed as inter-particle distance of Au NPs increased due to a broader size distribution.

ABSTRAK

Dalam kerja penyelidikan ini, kawalan saiz nanozarah emas (Au) yang disediakan pada filem silicon suboxide (SiO_x) telah dikaji dengan menggunakan teknik pemendapan wap. Bahagian pertama kerja ini melibatkan penyiasatan kesan keadaan persekitaran pada sifat-sifat struktur nanozarah emas. Teknik RF magnetron bersama-pemercikan telah digunakan untuk menyediakan filem Au/ SiO_x yang mempunyai struktur "dilonggokkan di atas" dan "tertanam dalam". Sampel yang baru disediakan telah disepuh lindap pada suhu yang berbeza untuk mengkaji kesan suhu sepuhlindap atas pertumbuhan nanozarah emas. Bagi kedua-dua struktur filem Au/ SiO_x , saiz dan bentuk nanozarah emas bergantung pada suhu. Nanozarah emas yang tertanam muncul dan mengedar dengan seragam pada permukaan filem SiO_x pada 800 °C. Manakala, nanozarah emas yang dilonggokkan di atas permukaan filem SiO_x menunjukkan penyebaran seragam zarah Au yang berbentuk memanjang dan pulau pada suhu 400 °C. Zarah Au telah berubah menjadi bentuk sfera apabila suhu penyepuhlindapan meningkat dari 400 hingga 800 °C. Pertumbuhan nanowayar SiO_x yang padat adalah mengikut pertumbuhan mekanisme pepejal-cecair-pepejal dan fenomena ini dapat diperhatikan pada suhu 1000 °C. Kedudukan dan lebar puncak permukaan plasmon resonans (SPR) amat bergantung pada saiz dan bentuk nanozarah emas. Untuk bahagian kedua kerja ini, hanya Au/ SiO_x dengan struktur "dilonggokkan di atas" telah dikaji kerana sifat morfologinya yang menarik selain daripada kaedah sintesis kawalan yang ringkas. Sistem pemendapan wap kimia secara peningkatan plasma (PECVD) buatan sendiri dan DC terbatuk-batuk coater telah diperkenalkan untuk menyediakan Au di atas filem SiO_x . Kesan nisbah kadar aliran gas $\text{N}_2\text{O}/\text{SiH}_4$ kepada pertumbuhan nanozarah emas telah dikaji dengan suhu sepuhlindap dikekalkan pada 800 °C. Nanozarah emas yang berkepekatan tinggi telah diagihkan secara sama rata pada permukaan filem SiO_x yang telah disediakan pada nisbah kadar aliran gas yang optimum, iaitu 30. FWHM bagi puncak SPR adalah faktor dominan yang berkorelasi dengan saiz zarah Au. Demi tujuan untuk mengurangkan pencemaran yang tidak diperlukan, dan berlaku semasa perpindahan sampel dari kebuk vakum ke DC terbatuk-batuk coater, proses satu langkah telah diperkenalkan – teknik Hot Wire bantuan PECVD. Penyejatan Au yang dikawal oleh pengatup dijalankan dengan serta merta di dalam ruang vakum selepas pemendapan filem SiO_x pada nisbah kadar aliran optima, iaitu 30. Peranan filem SiO_x sebagai penghalang untuk mencegah pengumpulan nanozarah emas telah dikaji. Ia mengesahkan bahawa interaksi kimia antara Au dan SiO_x yang lemah membolehkan kajian tepat ke atas nanozarah emas dengan mengelakkan faktor-faktor lain yang tidak diingini. Akhir sekali, kesan pemanasan substrat ke atas pertumbuhan nanozarah emas telah dikaji untuk menentukan kemungkinan untuk menyediakan nanozarah emas yang mempunyai saiz yang sekata dan lebih kecil tanpa menggunakan teknik terma-penyepuhlindapan akhir. Ia telah didapati bahawa nanozarah emas dengan saiz kira-kira 6.45 nm dapat diperoleh apabila suhu substrat ditetapkan pada 300 °C. Pembentukan nanozarah emas tidak dapat diperhatikan di bawah 300 °C. Proses penyepuhlindapan pada suhu yang lebih rendah (200 °C) ditunjukkan boleh menambah baik taburan nanozarah emas. FWHM bagi puncak SPR menjadi sempit apabila jarak antara nanozarah emas meningkat disebabkan oleh taburan saiz nanozarah Au yang lebar.

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LIST OF PUBLICATIONS

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- 1) **Keewah Chan**, Zarina Aspanut, Boontong Goh, Chornghaur Sow, Binni Varghese, Saadah Abdul Rahman, Muhamad Rasat Muhamad (2011), “Effects of post-thermal annealing temperature on the optical and structural properties of gold particles on silicon suboxide films”, *Applied Surface Science* 257, 2208-2213. I.F. 2.103
- 2) **Keewah Chan**, Zarina Aspanut, Boontong Goh, Muhamad Rasat Muhamad, Saadah A. Rahman (2011), “Formation of gold nanoparticles in silicon suboxide films prepared by plasma enhanced chemical vapour deposition”, *Thin Solid Films* 519, 4952-4957. I.F. 1.890
- 3) **Keewah Chan**, Boontong Goh, Saadah A. Rahman, Muhamad Rasat Muhamad, Chang Fu Dee, Zarina Aspanut (2012), “Annealing effect on the structural and optical properties of embedded Au nanoparticles in silicon suboxide films”, *Vacuum* 86, 1367-1372. I.F. 1.317
- 4) Najwa Rosli, **Kee Wah Chan**, Ilyani Putri Jamal, Saadah A. Rahman, Boon Tong Goh, Zarina Aspanut (2012), “Effect of Rapid Thermal Annealing Time on the Structural and Optical Properties of Layered Structured SiO_x/Au/SiO_x Film”. *Advanced Materials Research* 501, 221-225.
- 5) **Kee Wah Chan**, Boon Tong Goh, Saadah Abdul Rahman, Zarina Aspanut (2012), “Au/nc-Si:H Core-Shell Nanostructures Prepared by Hot Wire Assisted Plasma Enhanced Chemical Vapour Deposition Technique”, *Surface Coatings & Technology* *In Press, Corrected Proof* I.F. 1.867

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